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Certified by



Jon W Dudas

Acting Under Secretary of Commerce  
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13281 U.S. PTO  
07/23/03

# PROVISIONAL APPLICATION COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION under 37 CFR 1.53(c).

17849 U.S. PTO  
60/489111

07/23/03

Docket Number		34337-408		Type a plus sign (+) inside this box →	+
INVENTOR(s)/APPLICANT(s)					
LAST NAME	FIRST NAME	MIDDLE INITIAL	RESIDENCE (City and Either State or Foreign Country)		
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TITLE OF THE INVENTION (280 characters max)					
APPARATUS FOR MEASURING THE CRUSHING STRENGTH OF MICRON SUPERABRASIVES					
CORRESPONDENCE ADDRESS					
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STATE	Washington, D. C.	ZIP CODE	20005-3096	COUNTRY	USA
ENCLOSED APPLICATION PARTS (check all that apply)					
<input checked="" type="checkbox"/>	Specification	Number of pages [12]	<input type="checkbox"/>	Small Entity Statement	
<input type="checkbox"/>	Drawings	Number of sheets [0]	<input checked="" type="checkbox"/>	Other (specify): <u>Appendix A - 13 pages, Appendix B - 18 pages, Power Point presentation - 43 pages</u>	
METHOD OF PAYMENT (check one)					
<input type="checkbox"/>	A check or money order is enclosed to cover the Provisional filing fees			PROVISIONAL FILING FEE	
<input checked="" type="checkbox"/>	The Commissioner is hereby authorized to charge filing fees and credit Deposit Account Number: <u>500417</u>			AMOUNT (\$)	\$160.00

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

- ☒ No.
- ☐ Yes, the name of the U.S. Government agency and the Government contract number are:
- ☐ Additional inventors are being named on separately numbered sheets attached hereto.

Respectfully submitted,

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## **PROVISIONAL APPLICATION FOR PATENT**

### **Apparatus for measuring the crushing strength of micron superabrasives**

#### **Background of the Invention**

It is well known that fracture mechanism of superabrasive particles plays a determinant role in any abrasive process. The fracture mechanism of superabrasive particles is controlled by crystalline structure (i.e. monocrystalline vs. polycrystalline) and by the nature and concentration of crystal growth defects (i.e. pre-existing fractures, voids, mechanical stresses and impurities).

Generally, the sub-sieve powders (powders that are smaller than 400 mesh) are considered micron powders. However, in the size range from 40 microns (approx. 400 mesh) to 80 microns (approx. 200 mesh), the fine mesh sizes overlap with the coarse micron-sizes.

The characterization of micron superabrasive powders is a difficult and complex task, involving the evaluation of the properties of a very large number of particles. To date, the mechanical strength of micron superabrasive powders is controlled indirectly, by controlling the feed type and quality; i.e. metal bond diamond/cubic boron nitride (CBN) – belt press or cubic press synthesis process, as well as resin bond diamond/CBN – belt press or cubic press or opposed anvils press synthesis process.

#### **Summary of the Invention**

The present invention represents a breakthrough into the characterization of micron superabrasives (diamond and CBN powders) with respect to fracture strength (expressed

as crushing strength), a property that is ultimately responsible for their performance in most application areas.

The present invention describes an apparatus that measures the fracture strength of micron superabrasives, when subjected to crushing action under controlled conditions that replicate lapping process conditions.

The present invention describes an apparatus and the respective measurement technique used to characterize micron superabrasive powder types/products with respect to their fracture strength, expressed as crushing strength or resistance to crushing. The measurement technique is based on the evaluation of the ratio of particles that resisted crushing to the initial number of particles (before crushing) in a given micron superabrasive powder, when subjected to mechanical forces similar to those encountered in the lapping process.

The crushing strength technique makes possible direct and reliable comparisons between different micron superabrasives types/products with respect to their crushing strength.

#### **Detailed Description of the Invention**

The apparatus presented in Figure 1 uses the lapping process kinematics to crush micron superabrasive powders under controlled conditions, in size range down to 10 microns (mean size of the particle size distribution).

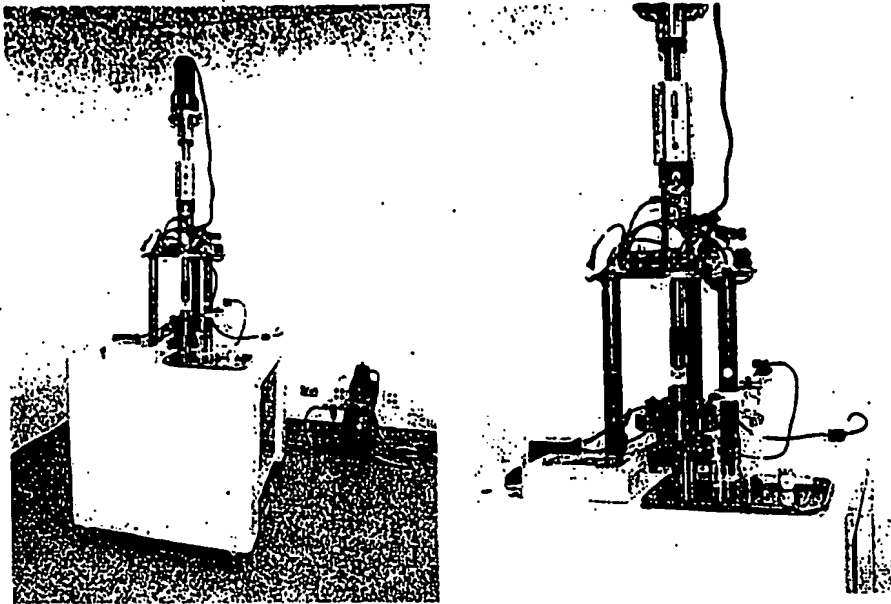
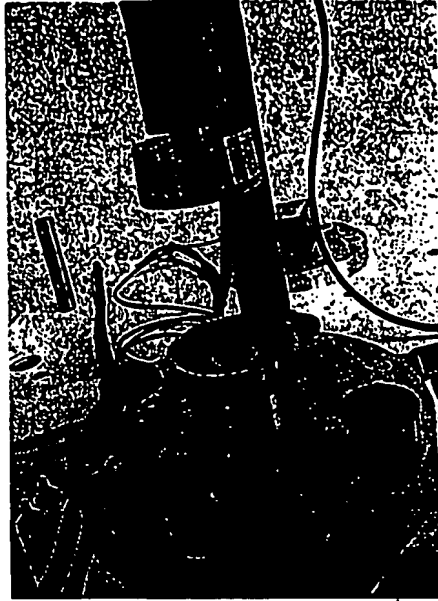


Figure 1 - Apparatus for testing the crushing strength of micron diamond powders

The main part of the apparatus is represented by the capsule for crushing the superabrasive powders, Figure 2, that consists of a steel cup and a steel piston that rotate independently in opposite directions at controlled speeds (rotations per minute - RPM). A pneumatic cylinder connected to the piston delivers the desired loads to the capsule. Both the face of the piston and the bottom of the cup are lined with polycrystalline diamond compact (PCD) discs to prevent contamination of the powders during crushing (through erosion of the steel parts), and, at the same time, to ensure precision and consistency of the test results. Thus, the micron superabrasive powders are crushed solely between the two PCD discs, without contact with the steel parts.



**Figure 2 – Test capsule and crushing strength apparatus**

Detailed drawings of the apparatus are presented in Appendix 1.

#### **Description of the crushing strength test**

A known amount (one carat) of micron superabrasive powder is charged into the cup on top of the PCD disc to form a circular layer of uniform thickness, Figure 3, and the crushing cycle is started.

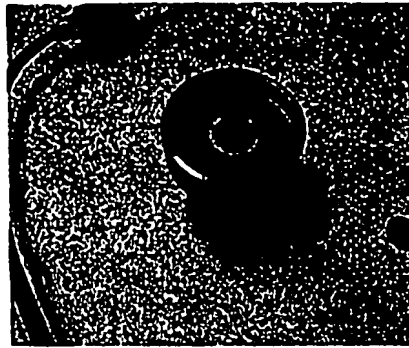


Figure 3 – Diamond powder charged into the test capsule

The crushing strength cycle is described below:

First, the piston is lowered into the capsule until it touches the superabrasive powder layer. Next, the desired load is applied. Following a short period of time allowed for the load to be evenly applied over the entire area under the piston in contact with the superabrasive particles, both the cup and the piston are rotated simultaneously in opposite directions with the desired speeds, for the entire duration of the crushing cycle. After completion of the crushing cycle, the rotation of the cup and the piston is simultaneously stopped, the load is removed and the piston is lifted.

The as-crushed micron superabrasive powder is then carefully collected into a glass beaker, by washing the cup and the piston with de-ionized (DI) water.

Prior to running crushing strength tests, the apparatus is calibrated with respect to applied load and the RPM of the cup and piston. The rotation speeds of both the cup and the piston can be varied from few RPM to 70 RPM.

To determine the fracture (crushing) strength of micron diamond/CBN powders, the particle size distributions (frequency distributions) of the original powders, as well as, of

the resulting powders, are measured using a particle size distribution (PSD) analyzer (i.e. Elzone 5382 analyzer). The PSD data are used to calculate the crushing strength index of the micron powders.

The following test parameters are introduced to quantify the crushing strength of micron superabrasive powders:

*On-size particles in the starting powder (OSS):* The cumulative percentiles of particles between 50% and 95% of the frequency distribution, in the starting superabrasive powder (prior to crushing),

*On-size particles in the resulting powder (OSR):* The cumulative percentiles of particles between 50% and 95% of the frequency distribution, in the resulting superabrasive powder (after crushing).

The crushing strength and crushing strength index were then defined as follows:

*Crushing strength:* The ratio of the on-size particles in the resulting powder (particles that resisted crushing), to the on-size particles in the starting powder,

*Crushing strength index (CSI):*

$$CSI = OSR/OSS \times 100$$

## **Experimental Results**

### **Measurement precision**

The precision of the crushing strength measurement was determined by running 5 crushing experiments in a row under constant test conditions (Load = 13.4 lb; time = 1 min; RPM = 10), on 10-20  $\mu$  MA, so-called "master diamond". The product designations "MA", "RA" and "IG" used herein are explained in Appendix B (see, e.g., Table 2 on page 6 of Appendix B). The arithmetic mean and standard deviation for on-



size particles in as-crushed powder (50% to 95% of the frequency distribution), as well as, the crushing strength index, were calculated. The experimental results are presented in Table 5.

Table 5 – Crushing strength measurement precision – 10 -20  $\mu$  MA

	Ref.	#1	#2	#3	#4	#5	Mean	St. Dev.
50%-95%	44.277	23.351	21.914	23.146	22.603	22.881	22.779	0.500296
CSI		52.7	49.5	52.3	51.0	51.7	51.4	1.1299

The above experimental data show that the crushing strength index of “master diamond” 10-20  $\mu$  MA is 51.4 +/- 1.1%, which indicate that the precision of the crushing strength measurement is better than 1.5%.

#### Examples of crushing strength tests

The results of crushing strength tests, on different micron diamond powder types/products are presented below (figures 4 to 6). In each case

#### Example 2

Crushing strength index (CSI) of 10-20 microns MA vs. IG vs. RA – Figure 4.

Test conditions:

Load = 13.4 lb;

Cup rotation speed = 10 rpm;

Head rotation speed = 10 rpm;

Crushing time = 30 sec

### **Example 2**

**Crushing strength index (CSI) of 10-20 microns MA vs. IG vs. RA – Figure 5.**

**Test conditions:**

**Load = 13.4 lb;**

**Cup rotation speed = 10 rpm;**

**Head rotation speed = 10 rpm;**

**Crushing time = 60 sec**

### **Example 3**

**Crushing strength index (CSI) of 10-20 microns MA vs. IG vs. RA – Figure 6.**

**Test conditions:**

**Load = 13.4 lb;**

**Cup rotation speed = 10 rpm;**

**Head rotation speed = 10 rpm;**

**Crushing time = 120 sec**

MA	54.3
IG	52.5
RA	40.7

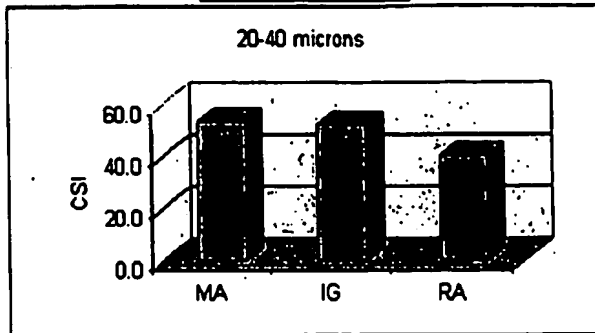


Figure 4 - Crushing Strength Test - 20-40 □ MA vs. IG vs. RA

MA	51.4
IG	43.4
RA	45.6

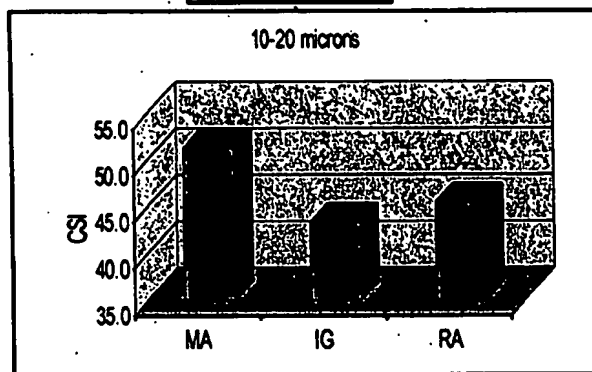


Figure 5 - Crushing Strength Test - 10-20 □ MA vs. IG vs. RA

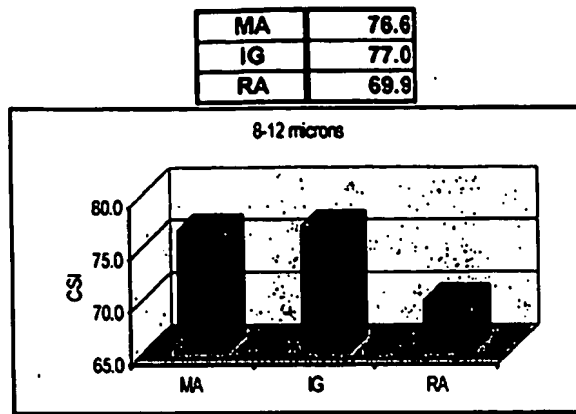


Figure 6 - Crushing Strength Test – 6-12 □ MA vs. IG vs. RA

As expected, the crushing strength data indicate that, under the test conditions employed, the metal bond diamond type/product synthesized via “belt press process” – MA, exhibits higher crushing strength than the resin bond diamond type/product synthesized via “opposed anvil press” process – RA, over the entire size range investigated. By contrast, the metal bond diamond type/product synthesized via “cubic press process” – IG, shows inconsistent crushing strength over the same size range and under same test conditions. While for 30 microns and 10 microns diamond powders the crushing strength of IG is similar to MA, the crushing strength of the 15 microns IG diamond powder is much lower than that of MA and almost equal to that of RA.

To date, a plethora of micron superabrasives types/products are available on the market, having one common denominator: “sub-sieve size”. The possibility of measuring (testing) the fracture strength of different micron superabrasive powders (diamond and CBN) enables the producers (micronizers) to better control the mechanical strength of their micron superabrasive types/products and, at the same time, enables the end users to

make an informed decision on selecting the micron type/product that performs best in each particular application, regardless if in loose abrasive, slurry, compound or bonded tool form.

**We claim:**

- 1. A method for measuring the crushing strength of an abrasive used in a lapping process, the abrasive comprising particles, the method comprising:**

  - determining an initial particle size distribution for the particles;**
  - subjecting the abrasive to a crushing force approximately equal to that of the lapping process;**
  - determining a post-crushing particle size distribution for the particles; and**
  - comparing the initial and post-crushing particle size distributions.**
  
- 2. An apparatus for measuring the crushing strength of an abrasive used in a lapping process, the abrasive comprising particles, the apparatus comprising:**

  - a cup for holding the abrasive;**
  - a first motor for rotating the cup in a first direction;**
  - a piston having a face for rotatably fitting within the cup and contacting the abrasive;**
  - a second motor for rotating the piston in a second direction opposite the first direction; and**
  - a press for pressing the piston against the abrasive and crushing the particles while the first and second motors are rotating.**

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